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PATENT SPECIFICATION 1,142,719

NO DRAWINGS

Inventors:—AVRAGHAM MATITIAJU BANIEL and RUTH BLUMBERG

1,142,719

Date of Application and filing Complete Specification: 5 Aug., 1966. No. 48996/67.

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Index at Acceptance:—C1 A11.

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COMPLETE SPECIFICATION

Phosphoric Acid Recovery.

ERRATUM

SPECIFICATION NO. 1,142,719

Page 1, Heading Inventors:- *for* "AVRAGHAM MATITIAJU BANIEL" *read* "AVRAHAM MATITIAHU BANIEL"

THE PATENT OFFICE,
30th April 1969 D 113838

containing phosphoric acid. The invention may be used for extracting phosphoric acid from a "technical-grade phosphoric acid", i.e. an aqueous phosphoric acid, usually manufactured by the decomposition of rock phosphate with sulphuric acid, having an H_3PO_4 concentration of not less than 35% by weight and possibly up to about 90% by weight, and containing the usual impurities of such phosphoric acid.

Our co-pending Application No. 10235/65 (Serial No. 1,112,033) claims a process for the extraction of phosphoric acid from an aqueous solution which comprises treating the solution with an extracting solvent capable of extracting phosphoric acid from an aqueous solution thereof having a concentration of H_3PO_4 above a threshold value of 35% by weight, whose capacity for extracting phosphoric acid from water varies inversely with temperature and which does not extract phosphoric acid from an aqueous solution thereof having a concentration below a threshold value of 35% by weight, the temperature of the treatment being low enough for the formation of a clear homogeneous extract phase distinct from a residual aqueous phase, separating the extract phase from the aqueous phase, raising the temperature of the extract phase to that at which it separates into a lower layer containing phosphoric acid and an upper layer composed mainly of solvent, and separating the layers, and further claims a solvent which (i) is capable of extracting phosphoric acid from an aqueous solution thereof having a concentration above a threshold value of 35% by weight, (ii) has an extracting power which varies inversely with temperature (that is, the solubility of phosphoric acid in the solvent is greater the lower the temperature, and the extracting power of the solvent falls with rising temperature) and (iii) it is not capable of extracting phosphoric acid from an aqueous solution having a concentration below a threshold value of 35% by weight.

The threshold value is the concentration of acid in water below which the solvent does not preferentially dissolve the acid from the water. The threshold value is not an exact figure, since it can vary with the temperature, and it is different for different solvent, but for the solvents to be used in this process it is about 35% by weight.

To make the process work using a solvent of characteristics (i), (ii) and (iii), one must effect the extraction at a relatively low temperature and then, taking advantage of the inverse temperature dependence of the extracting power, separate the extract phase into phosphoric acid and solvent at a temperature which is higher than the extracting temperature after removal of the extract phase from the aqueous solution.

The present invention is an improvement in or modification of the process of our said Application No. 1,112,033, and is concerned with specific solvents useful in carrying out the process of our said earlier application.

SEE ERRATA SLIP ATTACHED

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Phosphoric Acid Recovery.

We, ISRAEL MINING INDUSTRIES—INSTITUTE FOR RESEARCH AND DEVELOPMENT, an Israeli body corporate of Near Irganim, Haifa Bay, Israel, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to the recovery of phosphoric acid from an aqueous solution containing phosphoric acid. The invention may be used for extracting phosphoric acid from a "technical-grade phosphoric acid", i.e. an aqueous phosphoric acid, usually manufactured by the decomposition of rock phosphate with sulphuric acid, having an H_3PO_4 concentration of not less than 35% by weight and possibly up to about 90% by weight, and containing the usual impurities of such phosphoric acid.

Our co-pending Application No. 10235/65 (Serial No. 1,112,033) claims a process for the extraction of phosphoric acid from an aqueous solution which comprises treating the solution with an extracting solvent capable of extracting phosphoric acid from an aqueous solution thereof having a concentration of H_3PO_4 above a threshold value of 35% by weight, whose capacity for extracting phosphoric acid from water varies inversely with temperature and which does not extract phosphoric acid from an aqueous solution thereof having a concentration below a threshold value of 35% by weight, the temperature of the treatment being low enough for the formation of a clear homogeneous extract phase distinct from a residual aqueous phase, separating the extract phase from the aqueous phase, raising the temperature of the extract phase to that at which it separates into a lower layer containing phosphoric acid and an upper layer containing solvent, and separating the lower layer from the upper layer. It will be seen, then, that a solvent useful in the process of that application has the following characteristics: (i) it is capable of extracting phosphoric acid from an aqueous solution thereof having a concentration of H_3PO_4 above a "threshold value" of 35% by weight, (ii) its capacity for extracting phosphoric acid from water varies inversely with temperature (that is, the solubility of phosphoric acid in the solvent is greater the lower the temperature, and the extracting power of the solvent falls with rising temperature) and (iii) it is not capable of extracting phosphoric acid from an aqueous solution having a concentration below a threshold value of 35% by weight.

The threshold value is the concentration of acid in water below which the solvent does not preferentially dissolve the acid from the water. The threshold value is not an exact figure, since it can vary with the temperature, and it is different for different solvent, but for the solvents to be used in this process it is about 35% by weight.

To make the process work using a solvent of characteristics (i), (ii) and (iii), one must effect the extraction at a relatively low temperature and then, taking advantage of the inverse temperature dependence of the extracting power, separate the extract phase into phosphoric acid and solvent at a temperature which is higher than the extracting temperature after removal of the extract phase from the aqueous solution.

The present invention is an improvement in or modification of the process of our said Application No. 1,112,033, and is concerned with specific solvents useful in carrying out the process of our said earlier application.

SEE ERRATA SLIP ATTACHED

According to the present invention, therefore, there is provided a process as claimed in claim 1 of our Specification No. 1,112,033 in which the aqueous solution is extracted with cyclohexanone, butyl acetate, or dibutyl ether of diethylene glycol, or in which the aqueous solution is extracted with diethyl ether and the heat-treatment for the separation of the phases is effected under superatmospheric pressure.

The invention will now be illustrated by the following Examples in which all parts and percentages are by weight:—

EXAMPLE 1
(cyclohexanone)

160 g. of wet process phosphoric acid containing 60.5% H_3PO_4 (by wt.) was stirred at 20°C. with 60 g. of cyclohexanone for ten minutes, then the stirring was stopped. The mixture was allowed to stratify into two liquid phases, which were separated. The aqueous phase (at the bottom) contained about 56% of the phosphoric acid as well as the greater part of the impurities of the original wet process acid. The solvent phase containing the balance of the original phosphoric acid was heated at 65°C. and while being heated was mixed with about 15 ml. of water. Phase separation was thereby achieved. The top phase consisted of the solvent. The H_3PO_4 contained in the bottom was purified aqueous phosphoric acid of a concentration of 58%. This purified phosphoric acid was neutralized with ammonia to give a solution of diammonium phosphate without the formation of any precipitate.

EXAMPLE 2
(butyl acetate)

142 g. of wet process phosphoric acid containing 70% (by wt.) H_3PO_4 (about 100 g. H_3PO_4) was stirred at 2°C. with 40 g. of butyl acetate for ten minutes, then stirring was stopped. The mixture was allowed to stratify into two liquid phases and the latter were separated. The upper solvent phase contained 38 g. of H_3PO_4 (calculated as 100% H_3PO_4), the balance being in the lower phase together with the impurities of the original technical grade acid. The solvent extract phase was heated at 45°C. while being mixed with a small amount of water, in order to induce the phase separation. The resultant lower layer, which consisted of purified aqueous phosphoric acid of 65% H_3PO_4 concentration was separated and concentrated to 95% without deposition of solids during the evaporation.

EXAMPLE 3
(dibutyl ether of diethylene glycol)

200 ml. of wet process phosphoric acid containing 51% P_2O_5 were mixed at 25°C. with an equal volume of the dibutyl ether of diethylene glycol, resulting in an aqueous phase and 320 g. of solvent extract containing 140 g. H_3PO_4 (calculated as 100% H_3PO_4). This extract was decanted and heated to 100°C., a small amount of water being added to induce phase separation.

The bottom phase comprised 200 g. of purified phosphoric acid of 50% P_2O_5. This acid was concentrated to 95% H_3PO_4 without deposition of solids during evaporation.

EXAMPLE 4
(diethyl ether)

50 ml. of wet process phosphoric acid of 70% H_3PO_4 concentration were mixed at 25°C. with 100 ml. ethyl ether for 10 minutes obtaining 115 g. of extract containing 35% H_3PO_4. An aqueous phase containing 27% of the original P_2O_5, together with the impurities separated out at the bottom of the vessel.

The solvent extract removed was heated to 36°C. under pressure to obviate evaporation, thus inducing separation of the extracted P_2O_5 as a clean phosphoric acid.

WHAT WE CLAIM IS:—

1. A process for the extraction of phosphoric acid from an aqueous solution containing phosphoric acid, which comprises treating the aqueous solution with cyclohexanone, butyl acetate, dibutyl ether of diethylene glycol, or diethyl ether, the temperature of the treatment being low enough for the formation of a clear homogeneous extract phase distinct from a residual aqueous phase, separating the organic extract phase from the aqueous phase, raising the temperature of the extract phase to that at which it separates into a lower layer containing phosphoric acid and an upper layer containing solvent, said raising of the temperature being effected under superatmospheric pressure when the solvent is diethyl ether, and separating the lower layer from the upper layer.

2. A process as claimed in Claim 1, in which the temperature of the extract phase is raised by heating the extract to below its boiling point.

3. A process for the extraction of phosphoric acid from an aqueous solution therefore, substantially as described in Example 1, 2, 3 or 4.

4. Phosphoric acid obtained by a process as claimed in any one of claims 1 to 3.

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